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NOTES ON TWO FUNGICIDES: SULPHUR AND BORDEAUX MIXTURE

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"Wettable Sulphur."—As is well known through practice, sulphur kills those fungi known as "powdery mildews." Used dry in powder form, it has proved a cheap and reliable remedy against "mould" in hops, the mildews of the strawberry, rose, etc., and is in common use for this purpose. It not infrequently happens that crops which are affected with mildew suffer at the same time from damage caused by insects, and require, therefore, spraying with some insecticide, in which event a simultaneous application of sulphur and the insecticide is called for. In some cases, the need is met by using a dissolved compound of sulphur, such as "lime-sulphur" (calcium polysulphides) or "liver of sulphur" (potassium polysulphides), which can be mixed with the insecticide. In other cases, however, it is still advisable to use the sulphur in powder form, to avoid any injury being caused by the polysulphide solutions to the delicate parts of the plant sprayed. The question then arises of how to obtain an intimate mixture of sulphur powder with a wet spray.

It is common knowledge that it is impossible to mix sulphur powder with water, as it remains, even after the most vigorous stirring, on the surface. The use of soap as a medium for wetting sulphur appears to have been first proposed in 1910, by Vermorel and Dantony. They recommend* that sublimed sulphur be mixed with 1 per cent. soap solution and 1 per cent. sodium carbonate. As soap cannot be used satisfactorily when such spray-fluids as Bordeaux mixture, lime-sulphur, lead-arsenate, etc., are used, it was later recommended by the

* *Comptes Rendus*, Vol. 151, p. 1146 (1910).

same authors* that the sulphur should be moistened with an alcoholic solution of oleic acid, for sulphur so treated could at once be wetted with water. Alcohol alone suffices to make sulphur "wettable," but, when the alcohol evaporates, the sulphur again becomes unwettable. If, however, a small amount of oleic acid is incorporated with the sulphur, the latter retains the property of being easily wetted by water or spray-fluids. W. O. Gloyer,† in 1915, reported, from the results of experiments in the control of a stem-rot and leaf-spot of Clematis, caused by the fungus *Ascochyta clematidina*, that "a mixture of 1 lb. laundry soap and 6 lb. of sulphur to 15 gall. water" proved successful. It was also used with success in the control of leaf-blotch, *Diplocarpon rosae*, on roses grown in the forcing houses.

In order to obtain information as to the best, easiest and cheapest way of rendering sulphur wettable, a number of trials were carried out in the laboratory, in which the following substances were used: soft soap at various strengths, alcohol with and without oleic acid, oleic acid alone, casein in various forms, and glue. The practical points to which attention was directed were (*a*) ease of wetting; (*b*) the manner in which the sulphur remained in suspension; (*c*) the extent to which dilution could be carried out. It is not necessary to enter into details concerning the results which were obtained in the above trials, for it was found that a 1 per cent. solution of soft soap (10 lb. to 100 gal. of ordinary tap water of a hardness of 18°) proved most satisfactory in rendering the sulphur wettable, and that, with the water in question, dilution to ¼ per cent. of soap was possible without any separation of the sulphur.

Various brands of sublimed sulphur and ground sulphur were tested; all of them settled to the bottom of the vessel rather rapidly and could only be kept in suspension by constant agitation. Fine dusting sulphur, about 90 per cent. of which passed a 200-mesh sieve, was the most satisfactory sample tested, but the best commercial brands of sublimed sulphur and ground sulphur also gave satisfactory results. Tests, in which the various brands of sulphur were compared, showed that the sulphur remained in suspension in the case of the diluted soap solution as satisfactorily as in any other case except lime-casein (calcium caseinate), but, as the latter is much more troublesome in mixing, it is not recommended.

* *l.c.*: Vol. 153, p. 194 (1911).

† New York Agric. Exper. Station *Bull.* 44 (1915).

The addition of nicotine, at the rate of 6 oz. per 100 gal. to the sulphur, wetted with soap solution and with the other preparations—oleic acid and calcium caseinate—gave no indication of any change, and, consequently, the use of nicotine with wetted sulphur can be recommended for trial.

Biological Experiments.—During June, 1926, experiments* were carried out, in an unheated greenhouse, with hop plants infested with "mould" (*Sphaerotheca Humuli*). Marked leaves bearing a number of patches of the mildew, densely powdery with the accumulation of spores (*conidia*), were sprayed with a thick suspension of sulphur, previously treated with oleic acid, in a 1 per cent. solution of soft soap.†

The mixture was sprayed on in a very fine mist, using an atomizer. The container of the atomizer was well shaken each time before the application of the spray. No difficulty was found in getting the suspension to pass the extremely fine aperture of the nozzle. Unsprayed leaves at the same node, bearing similar patches of mildew, served as "controls." By the next day after spraying, all or most of the upright branches (*conidiophores*) of the mildew, as well as the chains of spores which they bear, were seen to be collapsed; no further growth of the mildew took place, the patches remaining sterile until the end of the experiment (11 to 14 days). The same satisfactory results were obtained when a mixture of the same amount of sulphur (with no oleic acid) and $\frac{1}{2}$ per cent. of soft soap was used. The rapid collapse of the mildew was in striking contrast with the effect observable when sulphur in powder form (sublimed or finely ground sulphur) is dusted on a patch of mildew. Dry sulphur causes no immediate collapse of the upright spore-bearing branches, which are only slowly affected at the relatively few places where the particles of sulphur touch the fungus—a fact on which further observations will be found below. The explanation of the difference of action is probably due to the fact that, in the fluid wash,

* The full details of these experiments will be published elsewhere.

† The brands of sulphur used were those known on the market as Brandram's "flowers of sulphur" (sublimed sulphur), Fortress Brand of Hop Sulphur (ground sulphur), and Monro's Dry Dusting Sulphur. The suspension was made by wetting 100 grammes of sulphur moistened with 4 c.c. methylated spirit and 1 c.c. oleic acid, and of this treated sulphur, 5 grammes were taken and added to 100 c.c. of 1 per cent. soft soap solution. Attention is called to the fact that this amount of sulphur—equivalent to $\frac{1}{2}$ lb. sulphur to the gallon—is not sprayed on to the plant, as a considerable proportion remains behind as a deposit. The amount of deposit varies according to the thoroughness of the agitation.

particles of sulphur are carried to all the parts of the fungus that are wetted, whereas only the surfaces of mildew patches are touched by the dry sulphur. Some observations which were made on hop leaves in the open, which were sprayed with the wettable sulphur, made as described above, incline us to believe that the sulphur particles remaining on the surface of the leaf, after the wash has dried, are less liable to be washed off by rain or dew than when sulphur is applied dry.

The sulphur particles suspended in the wash settle rapidly, as already pointed out, and constant agitation is required to maintain the suspension at the nozzle. It remains to be seen whether the agitation provided in the ordinary knapsack sprayer and in the horse-drawn hop-washer will be sufficient. The simultaneous application of sulphur and of an *aphis*-killing wash (nicotine, with or without soft soap) will, it is believed, prove feasible, and it is intended to carry out field experiments on these lines in the coming season.

Since it is the practice of some of the most successful hop-growers to apply sulphur in powder form to the hop plants when they are in flower (or, as the growers say, in "burr"), it was important to ascertain whether the application of a wash, containing wetted sulphur, would affect injuriously the flowers or the production of the hop cones. On August 6, 1926, a number of opposite lateral branches, which were in full "burr," on plants in the Experimental Hop-garden at Wye College, were selected, and one branch was heavily sprayed, an atomizer being used, with the wetted sulphur and soft soap solution described above, while the other branch was left unsprayed and served as a control. No injury resulted from the spraying, and, by the end of the month, it was found that the development of the cones on the sprayed and unsprayed branches was the same.

"*Flowers of Sulphur*" (*Sublimed Sulphur*) *and Ground Sulphur*.—In a previous communication to this JOURNAL,* one of the writers, when pointing out that sulphur is effective in controlling hop "mould" (*Sphaerotheca Humuli*), recommended for the purpose pure sublimed sulphur ("flowers of sulphur")—the statement being made "that flowers of sulphur remains the one safe and reliable remedy." This statement now requires modification. Within recent years, the use of improved machinery and the adoption of special apparatus have resulted in the manufacture of ground roll

* E. S. Salmon. This JOURNAL, XXVIII, p.157 (1921).

or brimstone sulphur which, in the case of good samples, is not inferior, in fineness and purity, to the best "flowers of sulphur."

During 1926, some experiments were carried out to compare the efficacy of sublimed sulphur and ground sulphur as fungicides against hop "mould." The method of applying the sulphur was as follows: a test-tube, 5 in. long and $\frac{9}{16}$ in. bore, was half-filled with the sulphur and its mouth covered with fine muslin. Sufficient sulphur was shaken out in a fine cloud to cover completely the powdery patches of mildew on hop leaves attached to a growing plant.

The first experiment was made on March 18, when similar patches of mildew, on two young leaves at the same node on the stem of a young hop plant, were treated with sublimed sulphur and the finest ground sulphur respectively. On April 1, 14 days after the application, the two leaves were removed and examined under the microscope. The fungicidal action of the two kinds of sulphur was the same. Wherever any particle of sulphur had touched the chains of spores borne on the upright branches (*conidiophores*) the former were shrivelled and dead, although the conidiophore below the chain of spores was uninjured; where, however, as at the edge of the leaf, no sulphur had touched the mildew, the latter was still vigorous and producing chains of spores. The sulphur was then removed as far as possible by washing the leaves; in the hollows near the veins of the leaves, the mildew had been protected from contact with the sulphur and was unaffected; elsewhere, the sulphur had killed the tallest branches (*conidiophores*) and their chains of spores, while the shorter, sheltered ones, not having touched the sulphur, were unaffected. There were no signs that the mycelial hyphae had been affected.

The second experiment, using the same two kinds of sulphur, was made on March 25, on two leaves at the same node, each bearing several large powdery patches of mildew. On April 27, 33 days after the application, the effect of the two sulphurs was exactly similar; where the sulphur had touched them, the chains of spores had all shrivelled, although here and there a young conidiophore with a short chain of spores occurred which was unaffected. The hyphae of the mycelium on the surface of the leaf were white and apparently unaffected.

In the third experiment, the same brands of sulphur were used, air-dry and after having been passed through a 160-

mesh sieve. The powders were applied, in the same manner as before, on May 19. On June 11, 23 days after the application, the mildew patches were still completely covered by the two powders; when the surplus was shaken off from some of the patches, these still seemed, to the eye, well covered by the sulphur. Under the microscope, fine hyphae of the mycelium could be seen radiating from the edges of each patch. There was no evidence that any of the mycelium was dead. Although the chains of spores were shrivelled and dead where contact with the sulphur particles had occurred, fresh conidiophores were appearing in the intervening spaces between the particles. By June 16, 28 days after the application, the remaining patches, still completely covered by the sulphur, were alive and showed young living hyphae at the circumference; intermingled with dead and shrivelled chains of spores were young conidiophores pushing up, with, very occasionally, a mature spore at the tip. It was evident that if the sulphur were removed—such as might result from the action of heavy rains—the mildew patches would be able to renew their growth and produce spores. No difference in action between sublimed sulphur and ground sulphur was observable.

It would appear from the results of the above experiments that, while sulphur in powder form does effectively stop the dispersal of spores of the mildew and thereby checks the spread of the disease, it is very slow in fungicidal action, and there is always the risk that it may be removed by heavy rains. Even when sulphur remained undisturbed for a month on the mildew patches, the fungus, under the conditions of the experiment, was not killed. Since the fungicidal action appears to be identical in both cases, the hop-grower can be advised to use either sublimed sulphur or special ground sulphur of equal fineness and purity.

In view of the slowness of action of sulphur when applied as a dust, the fluid wash of wetted sulphur (described above), with its much quicker fungicidal action, merits a trial against hop "mould" and other "powdery mildews."

Home-made Bordeaux Mixture.—The fungicide known as Bordeaux mixture (made by mixing together copper sulphate solution ["bluestone"] and milk of lime) is second to none in preventing many of the plant diseases caused by fungi. In all countries, it has been in use, for many years past, by the progressive farmer who realizes the benefits which it brings. The best method of making Bordeaux mixture has been given

in articles in this JOURNAL, in 1908 and again in 1910.* These articles may be consulted for detailed information both as to the making of stock solutions of copper sulphate and of lime, and for the proper application of the mixture. In the agricultural literature of every country, the results of numerous experiments have been published which show that Bordeaux mixture may confidently be relied upon to prevent the attacks of most fungi on cultivated crops. To take one instance, Bordeaux mixture effectively controls the "scab" or "black spot" of the apple, as recent articles in this JOURNAL have shown.†

Satisfactory results, obtained by the use of home-made Bordeaux mixture against other fungus diseases, have also been recorded.‡

The difficulty which is often experienced in obtaining, at short notice, small quantities of good freshly burnt quicklime, suitable for making Bordeaux mixture, has undoubtedly been one reason why many growers have not used this spray or have chosen a manufactured article.§ Moreover, quicklime does not retain its properties under the usual conditions of storage, so that the grower has to obtain fresh supplies each time he wishes to spray. As is well known, quicklime becomes hydrated by absorption of moisture and then combines with the carbon dioxide of the air to form carbonate of lime, which is worthless for making Bordeaux mixture. When quicklime is slaked with water, the hydrate of lime is produced, and it is this substance that combines with the copper sulphate to produce the well-known gelatinous blue precipitate, characteristic of properly made Bordeaux mixture. It is true that, as pointed out in 1910,|| perfectly satisfactory stock solutions of lime can be made with little trouble, but the farmer who shows the necessary enterprise, to have at hand stock solutions

* E. S. Salmon: "Apple Scab" or "Black Spot" (this JOURNAL, XV, 191 (1908); "The Making and Application of Bordeaux Mixture" (*l.c.*, XVI, 793 (1910).

† N. B. Bagenal, W. Goodwin, E. S. Salmon and W. M. Ware: "Spraying Experiments against Apple Scab" (*l.c.*, XXII, 137 (1925); XXXIII, 38 (1926).

‡ E. S. Salmon: "Cherry Leaf Scorch" (*l.c.*, XIV, 334 (1907); *Idem*: "Potato Spraying for Farmers" (*l.c.*, XXIV, 265 (1917); *Idem* and H. Wormald: "Potato Spraying Experiments" (*l.c.*, XXVI, 71, 269 (1919).

§ Experience both in this country and abroad has shown that home-made Bordeaux mixture is superior in fungicidal qualities to the ready-made preparation in paste or powder form.

|| This JOURNAL, XVI, 793 (1910).

of the ingredients to make Bordeaux mixture, remains, unfortunately, the rare exception.

The object of the present communication is to call attention to a variation in the method of making Bordeaux mixture, which consists in the use of slaked lime (calcium hydrate or hydrate of lime) in the place of quicklime (calcium oxide). Hydrate of lime is now prepared in a high degree of purity on a commercial scale, and, by its use, a simplification of the method of making Bordeaux mixture becomes possible. Comparative laboratory experiments made at Wye showed that the appearance and nature of the precipitate, as well as the rate of settling, were almost identical in the case of Bordeaux mixture made with quicklime and hydrate of lime, respectively, up to the end of 6½ hours; after longer periods the differences were only slight. On theoretical grounds, there seems to be no objection to the employment of hydrate of lime, for this is the product which is obtained in the milk of lime which, having been prepared by adding water to quicklime, is ordinarily used for the preparation of Bordeaux mixture. The advantages which can be obtained by using commercial hydrate of lime are (1) a high degree of purity; (2) a product which can be stored for comparatively long periods without becoming seriously changed; (3) a more rapid preparation of the spray fluid, as no time is lost in slaking the quicklime.

Hydrate of lime is obtainable on the English market in the form of a fine white, dry powder, and good commercial samples contain not less than 95 per cent. of hydrate; when packed fresh in closed paper-lined bags, it does not become carbonated at all rapidly. The proper packing is essential, for, unless this is done, the hydrate changes to carbonate, and there are no indications (as is the case with lumps of quicklime, which fall to a powder and so show that some change has taken place) to call attention to material which is no longer fresh and suitable for making Bordeaux mixture. As 1·32 parts by weight of hydrate of lime are equal to 1 part of oxide of lime, it is necessary to modify the proportions of copper sulphate and lime when the hydrate is used. A safe rule is to take 1½ pounds of the hydrate in place of 1 pound of quicklime, the usual formula for Bordeaux mixture, 8.8.100 thus becoming 8.12.100. The cost of the hydrate of lime will, of necessity, be somewhat higher than that of quicklime, especially if the latter can be obtained locally; but the advantages offered by the former are that it is a high-grade

product of uniform composition and, if kept closed in paper-lined bags, it does not change on storage. If the use of hydrate of lime becomes general, no doubt supplies will be obtainable in paper-lined bags of various sizes; at present, 56 lb. is the usual weight. The price of hydrate of lime, on rail at the works, is about £2 per ton, in four-ton lots, bags extra. It will be understood that hydrate of lime, even when packed in closed paper-lined bags, will not keep indefinitely, but from trials which were made last season it was found that it remained in a satisfactory condition from the time of the first spraying (April 17) to the last (June 19). The hydrate, when stored in closed metal containers, was fit for use after a lapse of nine months. It is recommended, however, that fresh supplies should be obtained for each season's spraying.

Reference to technical literature shows that in America hydrate of lime (slaked lime) has been in use for some time for making Bordeaux mixture. In a few cases, an adverse report has been given. Thus, Butler, in 1914, stated* that "hydrated lime does not produce as fine a milk of lime, all things equal, as quicklime, and the results [of experiments] show that this lack of fineness unmistakably increases the rate of settlement of the mixtures prepared with it. The use of hydrated lime, in lieu of quicklime, is not, therefore, to be recommended, except perhaps in the rare instances when the latter is not obtainable, or is of too poor quality to be advantageously used." In 1923, Martin carried out experiments in spraying potatoes against early blight (*Macrosporium Solani*), in which comparison was made of Bordeaux mixtures prepared from quicklime and from hydrated lime. This author states† that "it is apparent that the use of stone lime for making Bordeaux mixture is to be advised over that of hydrated lime. Where the former is difficult to secure, however, the hydrated lime may be substituted." In 1924, Newton, in reporting the results of spraying experiments against apple "scab," stated,‡ "The use of hydrated lime in Bordeaux mixture appeared to produce very much more severe russeting of the fruit than was caused by rock lime." Chupp, in his *Manual of Vegetable-Garden Diseases* (1925), states that "hydrated lime, if

* O. Butler: in New Hampshire Agric. Exper. Station *Circ.* No. 15 (May, 1914).

† W. H. Martin: "Ten Years of Potato Spraying in New Jersey," New Jersey Agric. Exper. Station *Bull.*, 383 (1923).

‡ Newton, F. W.: in *Trans. Illinois State Hort. Soc.*, LVII, 165 (1924).

fresh, may be employed if lump lime cannot be obtained, although Bordeaux mixture made of it does not stay in suspension quite as long as does that made from the burnt lime."

On the other hand, by far the greater number of experimenters report favourably on the use of hydrated lime in the preparation of Bordeaux mixture. Thus, Fields and Elliott, in 1920, stated* that "the hydrated lime Bordeaux mixture compared favourably with the mixture made from freshly slaked lime." In 1923, Winston, Bowman and Yothers reported† that "in general effectiveness Bordeaux mixture made with hydrated lime has proved throughout the experiments to be equal to that made with quicklime." Ballou and Lewis, in 1923, in Ohio, found‡ that a satisfactory control of apple "scab" was obtained by Bordeaux mixtures of various strengths, in which three pounds of high-grade hydrated lime were used for every pound of copper sulphate. Anderson, also, in 1924, in Illinois, in reporting on experiments in spraying apples against "scab," stated§ that Bordeaux mixture made with hydrated lime gave as good control and caused no more injury than Bordeaux mixture made from rock lime. In the *Spraying Programs for the Orchard and Fruit-Garden*, published by the Ohio Agricultural Experiment Station in 1920 and in 1922, the use of hydrate of lime is recommended.‖ Further references to authors advocating the use of hydrated lime are given below.¶

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* W. S. Fields and J. A. Elliott: in Univ. Arkansas Agric. Exper. Station *Bull.*, 172 (1920).

† J. R. Winston, J. J. Bowman, and W. W. Yothers: United States Dept. Agric. *Bull.*, 1178 (1923).

‡ F. H. Ballou and I. P. Lewis: in Ohio Agric. Exper. Station *Monthly Bull.*, VIII, 3-4, p. 42 (1923).

§ H. W. Anderson: in *Trans. Illinois State Hort. Soc.*, LVII, 165 (1923).

‖ *Monthly Bull.*, Ohio Agric. Exper. Station, V. No. 3, 70 (1920); VII, 1 and 2, 19 (1922).

¶ Green, Selby and Gossard, in Ohio Agric. Exper. Station *Bull.* 309 (1917); Halligan, Pettit and Coons, in *Sixty-First Annual Report*, State Board Agric., Michigan, 423 (1923); Robinson, in Oregon Agric. College Exper. Station *Bull.*, 201 (1924); Coons and Kotila, in *Sixty-Third Annual Report*, State Board Agric., Michigan, 505 (1925); Dutton, Pettit, Bennett and Cardinell, in Michigan Agric. College Agric. Exper. Station *Special Bull.*, 140 (1925); Dodge and Wilcox, in U.S. Dept. Agric. Farmers' *Bull.*, 1488 (1926); Young, Houser and Ballou, in Ohio Agric. Exper. Station *Bull.*, 393 (1926); and Manuel, in Australia, *Agric. Gaz.*, N.S. Wales, XXXIII, 759 (1922).

Robinson has recommended,* in the making of Bordeaux mixture with hydrated lime, the addition of 0·01 casein. The proportionate amount to be taken of hydrated lime as compared with quicklime is given by the various authorities quoted above (usually) as $1\frac{1}{2}$ lb. to 1 lb., although the ratios $1\frac{1}{3}$ to 1 and $1\frac{3}{4}$ to 1 are also given.

A comparative trial of Bordeaux mixtures made with quicklime and with hydrated lime, respectively, was carried out at Wye during 1926. Eight pounds of copper sulphate to 100 gal of water were used, and 8 and 12 pounds, respectively, of quicklime and hydrated lime. The latter was gradually mixed to a thin cream with cold water, and then diluted fully with the requisite amount of water before the concentrated copper sulphate solution (at the rate of 1 lb. of copper sulphate to 1 gal. of water) was poured into it. The same method of mixing was followed in making the Bordeaux mixture with quicklime. The two mixtures were sprayed on to an equal number of apple trees, of the varieties Newton Wonder and Allington Pippin, in a plantation on Wye College Farm. Three applications, using a fine, "misty" spray, were given; the first (at the "pink-bud" stage) on April 17; the second and third on May 21 and June 19. It was found that the Bordeaux mixture made with the hydrate of lime adhered to the foliage as well as that made with quicklime. Owing to a failure of the crop on the trees, the results of the spraying on the control of "scab" on the fruit could not be ascertained. It was observed, however, that the two Bordeaux mixtures controlled to a marked and equal extent the attacks of the fungus on the leaves, while causing no appreciable "scorching" injury. As early as May 29, many of the leaves on the "control" (unsprayed) plots showed so severe an infestation that they were becoming "sooty" with the growth of the fungus, while the foliage of the sprayed trees was practically free from attack. This control of the "scab" on the leaves of both Newton Wonder and Allington Pippin was continued right through the season, while the disease continued to develop on the unsprayed plots.

If, as seems likely to be the case, Bordeaux mixture made with hydrate of lime proves equal in fungicidal powers to that made with quicklime, a demand will grow up for the two constituents of the mixture prepared in convenient form. If weighed amounts of copper sulphate and of hydrate of lime are sent out by the manufacturers, with suitable instructions

* R. H. Robinson: in *Indus. and Eng. Chem.*, XV, 941 (1923).

for mixing, the making of home-made Bordeaux mixture on farms in this country may yet become as common as it should be.

Summary.—(1) A method is described of making a fluid wash of "wetted" sulphur, with or without soft soap.

(2) Experiments, carried out in 1926, showed that a suspension of sulphur in a solution of soft soap kills the "powdery mildew" of the hop (*Sphaerotheca Humuli*). The fungicidal action of the sulphur in this wash was observed to be far more rapid and complete than when sulphur was applied in powder form.

(3) Finely ground sulphur was found to be equal in fungicidal power (against the hop "powdery mildew") to sublimed sulphur.

(4) A method is described of making Bordeaux mixture with hydrate of lime in the place of quicklime.

(5) Preliminary experiments, made in 1926, showed that Bordeaux mixtures made with hydrate of lime and quicklime, respectively, controlled to an equal extent the attacks of "scab" (*Venturia inaequalis*) on the foliage of apple trees (Newton Wonder and Allington Pippin), and did not cause any "scorching" injury.

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